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**MULTI-PLY BACKSEAMABLE FILM**
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- (56) Prior Art Documents  
GB 2221649  
US 4608302
- (57) Claim

1. A heat-shrinkable multilayer film comprising a heat sealing layer comprising a blend of from about 50% to about 95% by weight of a propylene-based copolymer and from about 5% to about 50% by weight of a homogeneous ethylene alpha-olefin copolymer having a density below about 0.90 g/cc.

9. A heat-shrinkable multilayer film as set forth in claim 1 further including at least one internal layer containing a polyamide, a copolyamide or blends thereof.

14. A heat-shrinkable multilayer film comprising an inner heat sealing layer comprising a blend containing from about 5% to about 50% by weight of a homogeneous ethylene alpha-olefin copolymer having a density of below about 0.90 g/cc and an outer heat sealing layer comprising a blend containing from about 5% to about 50% by weight of a homogeneous ethylene alpha-olefin copolymer having a density of below about 0.90 g/cc.

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PATENT REQUEST : STANDARD PATENT

I/We being the person(s) identified below as the Applicant(s), request the grant of a patent to the person(s) identified below as the Nominated Person(s), for an invention described in the accompanying standard complete specification.

Full application details follow:

[71/70] Applicant(s)/Nominated Person(s):

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[54] Invention Title:

Multi-ply backseamable film

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Basic Convention Application(s) Details:

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DATED this TWENTY FOURTH day of SEPTEMBER 1993

*Keith Collison*

a member of the firm of  
DAVIES COLLISON CAVE for  
and on behalf of the  
applicant(s)

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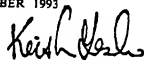
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NOTICE OF ENTITLEMENT

We, W. R. Grace & Co.-Conn., the applicant/Nominated Person named in the accompanying Patent Request state the following:-

The Nominated Person is entitled to the grant of the patent because the Nominated Person derives title to the invention from the inventor by assignment.

The Nominated Person is entitled to claim priority from the basic application listed on the patent request because the Nominated Person is the assignee of the applicant in respect of the basic application, and because that application was the first application made in a Convention country in respect of the invention.

DATED this TWENTY FOURTH day of SEPTEMBER 1993



.....  
a member of the firm of  
DAVIES COLLISON  
CAVE for and on behalf  
of the applicant(s)

(DCC ref: 1619569)

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**AUSTRALIA**  
**PATENTS ACT 1990**  
**COMPLETE SPECIFICATION**

**NAME OF APPLICANT(S):**

**W. R. Grace & Co.-Conn.**

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**INVENTION TITLE:**

**Multi-ply backseamable film**

The following statement is a full description of this invention, including the best method of performing it known to me/us:-

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#### FIELD OF THE INVENTION

The field of the invention is a packaging film from which bags and casings can be made which have improved structural soundness so that they may be fully characterized as "cook-in." The term "cook-in" as used herein is intended to refer to packaging material structurally capable of withstanding exposure to time-temperature conditions while containing a food product.

This invention relates generally to thermoplastic films suitable for cook-in packaging, and more particularly to food cook-in films. These films may have food contact surface characteristics which promote binding adherence to a contained food product during cook-in. The films of the invention exhibit improved hot and cold seal strength, improved optics and are more readily processable than known films. The present invention is particularly directed to backseamed casings.

#### PRIOR ART

Cook-in packaged foods are essentially foods cooked in the package in which they are distributed to the consumer and which may be consumed with or without warming. Cook-in time-temperature conditions typically refer to a long slow cook, for example, submersion in hot water at about 55°C to about 65°C for about 1 to about 4 hours. These conditions are repre-



sentative of institutional cooking requirements. Submersion at about 70°C to about 100°C for up to about 12 hours represents the limiting case. Under such conditions, a cook-in packaging material should maintain seal integrity, i.e., any heat sealed seams should resist being pulled apart during cook-in. The film should also be heat sealable to itself and, the packaging film substantially conformable to the contained food product. Preferably, this substantial conformability is achieved by the film being heat shrinkable under these conditions so as to form a tightly fitting package, i.e., the film should be heat shrinkable under these time-temperature conditions and should possess sufficient shrink energy so that submerging the packaged food product in hot water will shrink the packaging film snugly around the contained product, and especially up to about 55% monoaxial and/or biaxial shrinkage.

The film may also have food product adherence to restrict "cook-out" or collection of juices between the surface of the contained food product and the food contact surface of the packaging material during cook-in, thereby increasing product yield. More particularly, in the types of multilayer films wherein the first "sealing and food contact" layer is of the type of material that adheres to a contained food product during cook-in, this first layer may alternatively be referred to as the "adhering layer." As used herein, the term "adhere" is intended to mean that the food contact surface of the film bonds during cook-in to the contained food product to an extent sufficient to substantially prevent accumulation of fluids between the film and the contained product.

A heat shrinkable, cook-in film is described by Cberle et al, U. S. Patent No. 4,469,742 that includes a first "sealing or food contact" layer of a nonlipophillic polymeric material which has a softening point greater than that of the following shrink layer; a second layer, of an ethylene homopolymer or copolymer melt bonded to the first layer; a third or adhesive layer, melt bonded to the second layer, of a chemical-

ly modified polyethylene being irradiatively cross-linkable and having functional groups with a relatively strong affinity for the following barrier layer; a fourth or barrier layer of a hydrolyzed ethylene vinyl acetate copolymer melt bonded to the third layer; a fifth or adhesive layer as in said third layer, melt bonded to the fourth layer; and a sixth or abuse layer, melt bonded to the fifth layer. In one embodiment, the first "sealing and food contact" layer is an ionomer, a metal salt neutralized copolymer of an olefin and a carboxylic acid, representatively Surlyn(TM), which is a type of material that adheres to a contained meat product during cook-in. This Surlyn(TM) layer also functions as a protein-adhering layer. In another embodiment, the sealing layer is a propylene-ethylene random copolymer having from 1% to 5% by weight of ethylene. The patent also describes a method for making the film including full coextrusion and selective irradiation and orientation.

In the conventional method of manufacturing heat shrinkable film as described by Oberle et al, a tubular orientation process is utilized wherein a primary tube of the film is biaxially oriented by formation of a bubble to create internal pressure and induce stretching in the transverse direction and with the use of pinch rolls at different speeds to induce stretching in the machine direction. The films of the present invention are easily oriented with the incidence of bubble breakage during orientation minimized, and in some cases, eliminated. The stretched bubble is then collapsed, and may be wound up as flattened, seamless, tubular film to use later to make bags, e.g., either end-seal bags typically made by transversely heat sealing across the width of flattened tubing followed by severing the tubing so that the transverse seal forms the bag bottom, or side-seal bags in which the transverse heat seals form the bag sides and one edge of the tubing forms the bag bottom. Such bags are typically used by placing the food product in the bag, evacuating the bag, and either heat sealing the bag mouth or gathering and applying a metal clip around the gathered mouth of the bag to form a seal.

Alternatively, the collapsed tube may be slit into a film or two, or more films, depending on the width desired. The film can then be folded longitudinally about a forming shoe with opposed edges being joined in a lap seal in order to form a backseamed casing. Backseamed casings are often desirable when process limitations preclude formation of an oriented tube or sufficiently small diameter for a given cook-in application. However, the lap seal of such a casing requires that the inner layer of the cook-in material (optionally an adhering layer) is heat sealable to the outermost layer. This required sealing compatibility is most easily achieved by providing inner and outer layers of identical chemical composition. However, the inner and outer layers need not be identical as long as their respective compositions are sufficiently compatible for forming seals which are delamination resistant under rigorous cook-in conditions.

Formation of a backseamed tube from a film may be by a continuous or an intermittent process. By the intermittent process the film is typically sealed to form a length of tubing required for a given end-use application and then cut transversely. As the film indexes forward and the sealing and cutting steps are repeated, a series of tubular casings of the desired diameter and length are provided. The casings may be clipped at one end for subsequent stuffing and clipping of the opposite end or they may be heat sealed and treated as bags produced from seamless tubing. Alternatively, an intermittent process may be employed for forming a continuous tube so long as the individually formed seals have at least some overlap. By the continuous process the entire length of the film is sealed to form a continuous tube for later clipping, stuffing and clipping.

Cook-in tubing, whether seamless or backseamed, may be shirred; that is, folded or pleated into a relatively short length for ease of handling during stuffing. Regardless of the method of manufacture, the bag or casing is then immersed in hot water at approximately the same temperature at which



the film was stretch oriented, typically about 160° to 205°F. (61° to 95°C), hot water immersion being one of the quickest and most economical means of transferring sufficient heat to the film to shrink it uniformly. Alternatively, the bag, film or casing may serve as a liner of a cooking mold.

A plastic, adhering cook-in package such as a casing is described by Schirmer, in U. S. Patent No. 4,606,912 as well as a method for enhancing yield of a cook-in packaged meat product. The method includes first providing an adhering cook-in container including a flexible thermoplastic envelope being substantially conformable to a contained meat product and having an inner meat-contacting surface of a selectively irradiated ionomer, then conforming the container about a selected meat product and cooking the packaged product, whereupon the inner surface of the envelope bonds to the meat product substantially to prevent cook-out of fluids.

A flexible plastic adhering cook-in package is described by Thompson in U. S. Patent No. 4,411,919, as well as a method for enhancing yield of cook-in packaged meat product. The method includes: providing an adhering cook-in package comprising a flexible plastic container substantially conformable to a selected meat product and having an inner meat product contacting surface of polymeric olefin. This container is subjected to an energetic radiation surface treatment in the presence of oxygen sufficient to cause the inner surface to adhere to the meat product during cook-in, the container having been formed from hot blown tubular film. The package is conformed about a selected meat product followed by cooking the packaged product, whereupon the inner surface adheres to the meat product to substantially prevent cook-out of fluids.

Erk et al, U. S. Patent No. 4,303,711, describes a tubular film consisting of biaxially stretched plastic material for packing and casing, paste type foodstuffs that are heated after packing or are packed in a hot fluid state. The

film is a mixture of approximately 50-99 parts by weight of at least one aliphatic polyamide and approximately 1-50 parts by weight of one or more members of the group consisting of an ionomer resin, a modified ethylene vinyl acetate copolymer and a modified polyolefin.

Erk et al, U. S. Patent No. 4,601,929, relates to a single layer of polyamide film for packing and casing food-stuffs in paste form, especially foodstuffs that are packed when hot or are subject to heat treatment after packing.

Ghiradello et al, U. S. Patent No. 4,568,580, relates to an article of manufacture for packaging food products comprising a first film section having at least one surface comprising a copolyamide obtained by random copolymerization of precursor monomers of at least two different polyamides. The article includes a second film section of a film having a surface comprising the aforesaid copolyamide and at least one heat weld between the copolyamide surfaces of said first and second film sections. The article is capable of withstanding heat treatment at temperatures from 70°C to 120°C for at least 10 minutes without suffering damage to the heat weld.

Oberle, U. S. Patent No. 4,855,183 discloses a cook-in film having a first food contact layer comprising a polyamide composition.

Oberle, U.S. Patent No. 4,762,748 describes a cook-in shrink film characterized by the presence of ethylene alkyl acrylate copolymer in core layer and abuse layers, in adhesive layers, or both. The sealing layer is selected from the group consisting of a propylene ethylene copolymer, an ionomer, a blend of a linear low density polyethylene and an ionomer, and a blend of a linear low density polyethylene and an ethylene acrylic acid copolymer.

Schirmer, U.S. Patent No. 4,448,792, describes a cook-in shrink bag having a sealing and food contact layer of a

propylene homopolymer or copolymer, preferably a propylene-ethylene copolymer of low ethylene content. A core layer adjacent the sealing layer is a blend of a propylene homopolymer or copolymer. Also included are a barrier layer, an abuse layer and adhesive layers sandwiching the barrier.

Oberle, U.S. Patent No. 4,879,124, describes a perforated cook-in shrink bag. Preferable materials for the sealing layer include ethylene-acrylic acid copolymers, ethylene methacrylic acid copolymers, ionomers and blends of such.

Schirmer, U.S. Patent No. 4,608,302, describes oriented films from propylene copolymers and unplasticized Saran which are suitable for use in the packaging of prepared food products. A core layer may comprise an ethylene vinyl acetate copolymer having a vinyl acetate content of from 10% to 12% by weight and a fractional melt index. The core layer is adjacent a sealing layer which is a propylene homopolymer or copolymer which may be a propylene ethylene copolymer.

Bekele, U.S. Patent No. 4,909,726, describes an impact resistant film for chub packaging. Various layers may contain polymeric materials of fractional melt index.

Mueller, U.S. Patent No. 4,977,012, describes a barrier stretch film with at least one interior layer which may comprise an ethylene vinyl acetate copolymer of fractional melt index.

Schirmer, U.S. Patent No. 4,997,112, describes a film for chub packaging which includes an olefin sealing layer and a core layer adjacent the sealing layer which comprises a polymeric material of high molecular weight and fractional melt index selected from the group consisting of high density polyethylene, low density polyethylene and ethylene vinyl acetate copolymer.

U.S. Patent No. 4,495,249 issued to Chya et al discloses a multi-layer laminate film with a core layer of a hydrolyzed ethylene-vinyl acetate copolymer, and two outer layers of a mixture of copolymer of ethylene and vinyl acetate and a copolymer of propylene and ethylene or linear low density polyethylene. The multilayer laminate film of the reference can be made heat shrinkable and has gas barrier properties.

U.S. Patent No. 4,132,457 (Yamada et al) discloses a container with an EVCH core, adhesive layers, and additional layers of e.g. polypropylene or ethylene propylene copolymer.

U.S. Patent No. 4,511,610 (Yataki et al) discloses a container with an EVCH core, adhesive layers, and additional layers of e.g. polypropylene or ethylene propylene copolymer.

U.S. Patent No. 4,403,667 issued to Christensen et al appears to disclose a retortable pouch having a linear low density polyethylene heat seal layer, a second layer of a blend of linear low density polyethylene and propylene ethylene copolymer, optional third, fourth, and fifth layers of propylene ethylene copolymer, a sixth layer of an anhydride modified polypropylene, a seventh layer of nylon, an eighth layer of EVCH and a ninth layer or nylon.

U.S. Patent No. 4,532,159 issued to Mueller discloses a shrink film having two skin layers each comprising e.g. a blend of polypropylene and ethylene propylene copolymer.

Also of interest is U.S. Patent No. 4,400,422 issued to Rosenthal et al which discloses a composite film having a biaxially oriented polypropylene based film (BCPP) laminated on at least one surface with a multilayer structure including a gas barrier layer of a hydrolyzed ethylene vinyl acetate copolymer and a layer adjacent to the base film, and a heat sealable outer layer which may be, for example, modified propylene/ethylene copolymer.

EPO Patent Application No. 0149321 discloses a heat shrinkable tubular film having a gas barrier layer of vinylidene chloride copolymer, outer layers of polyolefin such as ethylene propylene copolymer, polypropylene and mixtures thereof, at least one intermediate layer of for example a polyamide, and adhesive layers disclosed between any of the above layers.

U.K. Patent Application GB 2,139,943A discloses a multilayer, preferably coextruded fiber-layer heat sealable film having a surface layer of a heat sealable polymer such as linear low density polyethylene of LLDPE blended with other polymers such as EVA, an EVOH layer, and a layer of polypropylene. These layers can be bonded by polymeric adhesive tie layers.

U.K. Patent Application GB 2,321,649 discloses a composite film with improved heat shrinkable properties comprising a core layer of a copolymer or propylene and one or more alpha-olefins having a Vicat softening point of 70-110°C and two outer layers of a crystalline polypropylene resin having a melting point of 135-150°C.

EPO Patent Application No. 435498-A is directed to a multilayer heat-shrinkable film which includes a heat sealing layer which is a copolymer of propylene and at least two other alpha-olefins.

Japanese Patent Application 1051133-A is directed to a heat shrinkable multilayer film which includes at least one layer which is a blend of an aliphatic polyamide and a non-crystalline polyamide.

Japanese Patent Application 1121542-A is directed to a packaging material which contains an inner layer which is a polyamide/polyethylene blend.



German Patent Application 3943024-A is directed to a monolayer casing which is a blend of nylon 6 and a copolyamide.

### Summary of the Invention

According to the present invention there is provided a heat-shrinkable multi layer film comprising a heat sealing layer comprising a blend of from about 50% to about 95% by weight of a propylene-based copolymer and from about 5% to about 50% by weight of a homogeneous ethylene alpha-olefin copolymer having a density below about 0.90 g/cc.

According to a further embodiment, the present invention provides a heat-shrinkable multi layer film comprising an inner heat sealing layer comprising a blend containing from about 5% to about 50% by weight of a homogeneous ethylene alpha-olefin copolymer having a density of below about 0.90 g/cc and an outer heat sealing layer comprising a blend containing from about 5% to about 50% by weight of a homogeneous ethylene alpha-olefin copolymer having a density of below about 0.90 g/cc.

### Detailed Description of the Invention

The present invention is directed generally to multilayer heat shrinkable films which have a heat sealing layer which is a blend of a propylene based copolymer and a homogeneous ethylene alpha-olefin copolymer with a density below about 0.90 g/cc. Specifically, films with improved process-ability and heat seal characteristics in accordance with the present invention have the structure: (inner sealing and food contact layer) - (nylon layer) - (outer sealing layer) or (inner sealing and food contact layer) - (nylon layer) - (barrier layer) - (outer sealing layer). Here again, the inner sealing and food contact layer is a blend of a propylene based copolymer with an ethylene alpha-olefin copolymer having a density below about 0.90 g/cc. The propylene based copolymer is preferably an ethylene propylene copolymer which contains from about 94% to about 99% by weight of propylene and from about 1% to about 6% by weight of ethylene.



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Alternatively, the propylene based copolymer may be a copolymer of propylene with ethylene and butene. Such ethylene propylene butene copolymer should preferably contain from about 90% to about 99% by weight of propylene, from about 1% to about 6% by weight of ethylene, and from about 1% to about 10% by weight of butene. It should be noted that

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purposes of the present specification, the term "copolymer" is intended to broadly describe the products of polymerization of two or more comonomers.

Although the present heat sealing layer may comprise anywhere from 50% to about 95% by weight of such propylene based copolymer, a more preferred range is 70% to about 85% with 75% of the heat sealing layer being the optimum amount of such copolymer. As noted above, the propylene based copolymer is blended with a homogeneous ethylene alpha-olefin copolymer which has a density below about 0.90 g/cc.

Ethylene alpha-olefins are, generally speaking, copolymers of ethylene with one or more comonomers selected from C<sub>3</sub> to about C<sub>10</sub> alpha olefins but especially comprises ethylene copolymers with C<sub>3</sub> to about C<sub>10</sub> alpha olefins such as butene-1, pentane-1, hexene-1, octene-1, and the like, in which the polymer molecules comprise long chains with few side chains or branches and sometimes are referred to as linear polymers. These polymers are obtained by low pressure polymerization processes and the side branching which is present will be short compared to non-linear ethylenes. Ethylene/alpha-olefin copolymers have a density in the range of from about 0.860 g/cc to about 0.940 g/cc. The term linear low density polyethylene is generally understood to include that group of ethylene/alpha-olefin copolymers which fall into the density range of about 0.915 to about 0.940 g/cc. Sometimes linear polyethylene in the density range from about 0.925 to about 0.940 is referred to a linear medium density polyethylene (LMDPE). Lower density ethylene alpha olefins may be referred to as very low density polyethylene (VLDPE, typically used to refer to the ethylene butene copolymers supplied by Union Carbide) and ultra-low density polyethylene (ULDPE, typically used to refer to the ethylene octene copolymers supplied by Dow). It should be noted although specific density ranges is for VLDPE, ULDPPE, LLDPE, and LMDPE have been set forth herein, that no bright line can be drawn for density classification and such will vary by supplier.



Heretofore, conventional ethylene alpha-olefins have been produced by Ziegler-Natta catalyst polymerization. The traditional Ziegler-Natta homogeneous system includes a metal halide activated by a metal alkyl cocatalyst. These are systems in which there are various oxidation states of the metal, and a variety of ligand environments, depending upon where the site is located in the catalyst, all leading to a relatively broad molecular weight distribution and a generally heterogeneous composition. Generally speaking, Ziegler-Natta catalysts incorporate comonomer poorly and produce some polymer chains with much comonomer and others with substantially less. For example, with conventional Ziegler-Natta catalysis, a linear ethylene alpha-olefin having an average of 10% comonomer may have a range of 0% to 40% comonomer in any given individual chain.

Recently a new type of ethylene based linear polymers have been introduced. These new resins are produced by metallocene catalyst polymerization and are characterized by narrow or more homogenous compositional properties, such as molecular weight distribution, than resins produced by conventional Ziegler-Natta polymerization processes. Conventional Ziegler-Natta polymerization systems have discreet catalyst composition differences which are manifested as different catalyst reaction sites with each site having different reaction rates and selectivities. Metallocene catalyst systems are characterized as a single identifiable chemical type which has a singular rate in selectivity. Thus the conventional systems produce resins that reflect the differential character of the different catalyst sites while resins produced by metallocene systems reflect the single catalytic site. However, it should be noted that at least some previously available ethylene based linear polymers exhibited the physical and compositional properties achieved by the present metallocene catalyzed polyolefins. Although not produced by metallocene catalysis, such copolymers were produced by non-metallocene single site catalysts yielding resins which achieve the homogeneity of metallocene catalyzed resins. An example of such are

the resins sold under the trade name Tafmer(TM) by Mitsui. Both metallocene catalyzed ethylene alpha olefins and the Tafmer-type of resins are appropriate for use in the heat seal layer of the present invention.

Thus, the ethylene alpha-olefin of the present heat sealing layer is a homogeneous metallocene catalyzed resin or, at least, a single site catalyzed resin such as a Tafmer. The density is limited to below about 0.90 g/cc in order to aid i processability of the overall film structure and for improved sealing. The homogeneous ethylene alpha-olefin employed is preferably an ethylene butene copolymer or an ethylene propylene copolymer which will exhibit excellent compatibility with the propylene-based resin. <sup>In a preferred embodiment</sup> the ethylene alpha-olefin will comprise from about 5% to about 50% by weight of the heat sealing layer, most preferably between 15% and 30%.

The present film structure further includes at least one polyamide or nylon-containing layer. Such layer is present to impart elastic recovery properties to the overall structure. Specifically, in a cook-in application, as noted above, the food product is literally cooked in the film package. Because the present film is biaxially oriented it shrinks during cooking to closely conform to the enclosed food product. After cooking, the product is typically chilled which, at least in the case of a meat product, often results in product shrinkage. It is desirable that the film have good elastic recovery in order to tightly conform to the meat during such shrinkage.

It is generally known that nylons provide such elastic recovery properties to thermoplastic films. For purposes of the present invention, various combinations of polyamides, copolyamides and amorphous nylons were investigated for determining a preferred combination of processability, stiffness imparting properties and elastic recovery. A layer of nylon 6/12 was found to produce the optimum combination of properties for the present invention.

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Thus, the invention preferably comprises the foregoing (inner sealing and food contact layer)/(nylon layer)/(outer sealing layer) and in further embodiments the structure: (inner sealing and food contact layer)/(nylon layer)/(barrier layer)/(outer sealing layer). Each of the layers may also be optionally bonded to one another by a tie layer as well.

The food contact layer can optionally be subjected to an energetic radiation treatment, including, but not limited to corona discharge, plasma, flame, ultraviolet, and high energy electron treatment. For instance, the food contact layer may be selectively irradiated with high energy electrons which advantageously may be accomplished during irradiation of the overall multilayer film structure for cook-in integrity, as further discussed below. Radiation dosages are referred to herein in terms of the radiation unit "RAD," with one million RADS or a megarad being designated as "MR." A suitable radiation dosage of high energy electrons is in the range of up to about 12 MR, more preferably about 2 to about 9 MR.

The adhesive or tie layer will also comprise any polymer that will have excellent adhesion to the barrier layer and the core layer or the abuse layer or both the core layer and the abuse layer. The various polyethylenes including the polyethylene copolymers as described herein and the ethylene vinyl acetate copolymers are useful as tie layers employed according to the present invention. Preferably, the tie layer comprises an anhydride modified alpha-olefin copolymer having rubber moieties such as is contained in the core layer, discussed above. A preferred resin is Tymor 1203 from Morton International. Also suitable is Bynel 4107 from du Pont. However, any of the various adhesives well known in the art of film making may be employed, such as, for example, the Plexar (TM) adhesives supplied by Chemplex Co.

The term "ethylene vinyl acetate copolymer" (EVA) as used herein for a type of polyethylene refers to a copolymer formed from ethylene and vinyl acetate monomers wherein the



ethylene derived units in the copolymer are present in major amounts and the vinyl acetate derived units in the copolymer are present in minor amounts. EVA is known not only for having structural strength, as ethylene alpha-olefin polymer does, but also for providing excellent adhesion to an adjacent layer, which decrease or even obviate the need for an "adhesive." EVA copolymers can be hydrolyzed to ethylene vinyl alcohol copolymers, (EVOH), which are employed principally as a barrier layer according to the present invention. For enhanced orientability a modifier such as an ethylene/acrylic ester/maleic anhydride terpolymer may be blended with the present EVOH barrier layer.

The term "oriented" is also used herein interchangeably with the term "heat shrinkable," these terms designating a material which has been stretched and set by cooling while substantially retaining its stretched dimensions. An oriented (i.e. heat shrinkable) material will tend to return to its original unstretched (unextended) dimensions when heated to an appropriate elevated temperature.

The cook-in film of the present invention is preferably oriented and is formed by extrusion processes especially art-known coextrusion methods. It is initially cooled to by, for example, cascading water quenching, after which it is reheated to within its orientation temperature range and oriented by stretching. The stretching to orient may be accomplished in many ways such as, for example, by "blown bubble" techniques or "tenter framing." These processes are well known to those skilled in the art and refer to orientation procedures whereby the material is stretched in the cross or transverse direction (TD) and/or in the longitudinal or machine direction (MD). After being stretched, the film is quickly quenched while substantially retaining its stretched dimensions to cool the film rapidly and thus set or lock in the oriented molecular configuration.



If a film having little or no orientation is desired, e.g. nonoriented or non-heat shrinkable film, the film may be formed from a nonorientable material or, if formed from an orientable material may be "hot blown." In forming a hot blown film, the film is not cooled immediately after extrusion or coextrusion but rather is first stretched shortly after extrusion while the film is still at an elevated temperature above the orientation temperature range of the material. Thereafter, the film is cooled, by well known methods. Those having skill in the art are familiar with this process and the fact that the resulting film has substantially unoriented characteristics. Other methods for forming unoriented films are well known such as cast extrusion or cast coextrusion methods.

As noted, the film layers may be formed by coextrusion, with additional layers thereafter being extrusion coated thereon to form multilayer films. Two multilayer tubes may also be formed with one of the tubes thereafter being extrusion coated or laminated onto the other. The extrusion coating method of film formation is preferable to coextruding the entire film when it is desired to subject one or more layers of the film to a treatment which may be harmful to one or more of the other layers. This may be done where it is desired to irradiate one or more layers of a film with high energy electrons where the film contains a barrier layer of one or more copolymers of vinylidene chloride (e.g., Saran(TM)), such as vinylidene chloride and vinyl chloride or vinylidene chloride and methyl acrylate as well as vinylidene chloride with ethyl acrylate or acrylonitrile.

Films of this type would, for example, comprise those where the barrier layer is a Saran(TM) layer in addition to or instead of an EVCH layer. Those skilled in the art generally recognize that irradiation with high energy electrons is generally harmful to such Saran(TM) barrier layer compositions, as irradiation may degrade and discolor Saran(TM), making it turn brown. Thus, if full coextrusion followed by high energy

electron irradiation of the multi layer structure is carried out on a film having a barrier layer containing a Saran (TM) layer, the irradiation should be conducted at low levels and with care. Alternatively, this may be avoided by extruding a first layer or layers, subjecting the layer or layers to high energy electron irradiation and thereafter applying the Saran (TM) barrier layer and, for that matter, other layers (which may or may not have been irradiated) sequentially onto the outer surface of the extruded previously irradiated tube. This sequence allows for high energy electron irradiation of the first layer or layers without subjecting the Saran (TM) barrier layer to harmful discolouration.

10 Thus, as used herein the term "extrusion" or the term "extruding" is intended to include coextrusion, extrusion coating, or combinations thereof.

The above general outline for manufacturing of films is not meant to be all inclusive since such processes are well known as set forth in U.S. Patent Nos. 4,274,900; 4,229,241;  
15 4,194,039; 4,188,443; 4,048,428; 3,821,182 and 3,022,543.

Advantageously in certain embodiments the present invention provides a novel cook-in film which is readily biaxially oriented.

20 In further embodiments the present invention provides a novel cook-in film that has improved biaxial orientability as a tube by means of forming a bubble wherein bubble breakage is substantially minimised or eliminated.

In further embodiments the present invention provides a method for manufacturing  
25 such a novel cook-in film wherein the sealing layer comprises a blend of a propylene based copolymer and an ethylene alpha-olefin copolymer.

In further embodiments the present invention provides a method for manufacturing such a novel cook-in film wherein the outermost layer comprises a blend of a propylene based  
30 copolymer and an ethylene alpha-olefin copolymer.

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In yet further embodiments the present invention provides a cook-in film with good elastic recovery for a tight package appearance.

In still further embodiments the present invention provides cook-in films which can  
5 be formed into backseamed casings.

Additional advantages of the invention have been set forth in part in the description and in the examples which follow and in part will be apparent to a person with ordinary skill in the art from the description, or may be learned by practice of the invention. The  
10 advantages of the invention may be realized and attained by means of the instrumentalities and combinations particularly pointed out in the appended claims.

Throughout this specification and the claims which follow, unless the context requires otherwise, the word "comprise", or variations such as "comprises" or "comprising", will be  
15 understood to imply the inclusion of a stated integer or group of integers but not the exclusion of any other integer or group of integers.

The present invention will be further understood with reference to the following non-limiting examples and data.



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# EXAMPLES

An attempt was made to produce the following film structures. It was believed that the polyamide composition of the internal layers would provide good elastic recovery properties to a conventional inner sealant / tie / barrier / tie / outer sealant structure. However, these comparative film structures proved difficult and sometimes impossible to orient. Relative percent thicknesses for each layer are also given.

TABLE I

Layers	2	3	4	5	6	
Inner Sealant	Tie	Nylon	Barrier	Nylon	Tie	Outer Sealant
Comp. Ex. 1						
EPC-1	R-AD	PA(90%) APA (10%)	EVCH	PA(90%) APA (10%)	R-AD	EPC
7.35%	8.82%	7.35%	8.82%	29.41%	29.41%	9.5
Comp. Ex. 2						
EPC-1	P-AD	PA(90%) APA (10%)	EVCH	PA(90%) APA (10%)	P-AD	EPC
29.41%	8.82%	7.35%	8.82%	7.35%	8.82%	29.4
Comp. Ex. 3						
EPC-1	R-AD	PA(90%) APA (10%)	EVCH	PA(90%) APA (10%)	R-AD	EPC
31.75%	9.52%	3.97%	9.52%	3.97%	9.52%	31.7
Comp. Ex. 4						
EPC-1	P-AD	PA(90%) APA (10%)	EVCH-3	PA(90%) APA (10%)	R-AD	EPC
31.75%	9.52%	3.97%	9.52%	3.97%	9.52%	31.7



Comp.  
Ex. 5

EPC-1	R-AD	PA(85%) APA (15%)	EVCH PA(85%) APA (15%)	R-AD	EPC
11.75%	9.52%	3.97%	9.52%	3.97%	31.7

Comp.  
Ex. 6

EPC-1	R-AD	PA(85%) APA (15%)	EVCH(85%)PA(55%) M (15%)APA (15%)	R-AD	EPC
11.75%	9.52%	3.97%	9.52%	3.97%	31.7

Comp.  
Ex. 7

EPC-1	R-AD	PA(93%) APA (7%)	EVCH-2 PA(93%) APA (7%)	R-AD	EPC
11.75%	9.52%	3.97%	9.52%	3.97%	31.7

Comp.  
Ex. 8

EPC-1	P-AD	PA(90%) APA (10%)	EVCH-2 PA(90%) APA (10%)	P-AD	EPC
30.30%	11.36%	3.79%	9.09%	3.79%	30.3

Comp.  
Ex. 9

EPC-1	P-AD	PA(90%) APA (10%)	EVCH-2 PA(90%) APA (10%)	P-AD	EPC
31.50%	11.81%	1.97%	9.45%	1.97%	31.5

The following film structures, all containing a blend of a propylene-based copolymer and an ethylene alpha-olefin in the sealant were readily processed with little or no difficulty in orientation. It should be noted that the structures of the present Examples include polyamide-containing layers of comparable thicknesses to those of Comparative Examples 1 - 9 above. In those earlier structures the polyamide was split into two separate layers to sandwich the barrier layer and to provide a palindromic film structure. It was believed that a symmetrical structure would preclude curling. However, for the present Examples it was determined that the stiffness of the barrier layer balanced that of a single polyamide-containing layer to prevent curling.

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TABLE II					
1 Layers Inner Sealant	2 Tie	3 Nylon	4 Barrier	5 Tie	6 Outer Sealant
Ex. 1 EPC-1(75%) EAO-1(25%) 25.81%	R-AD	CPA-1(80%) APA (20%) 6.45%	EVCH(85%) M (15%) 9.68%	R-ADEPC-1(75%) EAO-1(25%) 16.13%	25.81%
Ex. 2 EPC-1(75%) EAO-1(25%) 24.24%	R-AD	CPA-1(80%) APA (20%) 12.90%	EVCH(85%) M (15%) 9.09%	R-ADEPC-1(75%) EAO-1(25%) 15.15%	24.24%
Ex. 3 EPC-1(75%) EAO-1(25%) 24.24%	R-AD	CPA-1(90%) APA (10%) 12.90%	EVCH(85%) M (15%) 9.09%	R-ADEPC-1(75%) EAO-1(25%) 15.15%	24.24%
Ex. 4 EPC-1(75%) EAO-1(25%) 25.81%	R-AD	CPA-1(90%) APA (10%) 6.45%	EVCH(85%) M (15%) 9.68%	R-ADEPC-1(75%) EAO-1(25%) 16.13%	25.81%
Ex. 5 EPC-1(75%) EAO-1(25%) 25.81%	R-AD	CPA-2(90%) APA (10%) 6.45%	EVCH(85%) M (15%) 9.68%	R-ADEPC-1(75%) EAO-1(25%) 16.13%	25.81%
Ex. 6 EPC-1(75%) EAO-1(25%) 24.24%	R-AD	CPA-2(90%) APA (10%) 12.90%	EVCH(85%) M (15%) 9.09%	R-ADEPC-1(75%) EAO-1(25%) 15.15%	24.24%
Ex. 7 EPC-1(75%) EAO-1(25%) 25.81%	R-AD	CPA-1(60%) CPA-2 (40%) 6.45%	EVCH(85%) M (15%) 9.68%	R-ADEPC-1(75%) EAO-1(25%) 16.13%	25.81%
Ex. 8 EPC-1(75%) EAO-1(25%) 24.24%	R-AD	CPA-1(60%) CPA-2 (40%) 12.90%	EVCH(85%) M (15%) 9.09%	R-ADEPC-1(75%) EAO-1(25%) 15.15%	24.24%
Ex. 9 EPC-1(75%) EAO-1(25%) 25.81%	R-AD	PA-1(60%) CPA-1 (40%) 6.45%	EVCH(85%) M (15%) 9.68%	R-ADEPC-1(75%) EAO-1(25%) 16.13%	25.81%
Ex. 10 EPC-1(75%) EAO-1(25%) 24.24%	R-AD	PA-1(60%) CPA-1 (40%) 12.90%	EVCH(85%) M (15%) 9.09%	R-ADEPC-1(75%) EAO-1(25%) 15.15%	24.24%

Ex. 11				
EPC-1(75%)	R-AD	PA-1(60%)	EVCH(85%)R-ADEPC-1(75%)	
EAO-1(25%)		CPA-2 (40%)	M (15%)	EAO-1(25%)
24.24%	15.15%	12.90%	9.09%	15.15%24.24%

Ex. 12				
EPC-1(75%)	R-AD	PA-1(60%)	EVCH(85%)R-ADEPC-1(75%)	
EAO-1(25%)		CPA-2 (40%)	M (15%)	EAO-1(25%)
25.81%	16.13%	6.45%	9.68%	16.13%25.81%

Ex. 13				
EPC-1(75%)	R-AD	PA-1(90%)	EVCH(85%)R-ADEPC-1(75%)	
EAO-1(25%)		APA (10%)	M (15%)	EAO-1(25%)
25.81%	16.13%	6.45%	9.68%	16.13%25.81%

Ex. 14				
EPC-1(75%)	R-AD	PA-1(90%)	EVCH(85%)R-ADEPC-1(75%)	
EAO-1(25%)		APA (10%)	M (15%)	EAO-1(25%)
24.24%	15.15%	12.90%	9.09%	15.15%24.24%

Ex. 15				
EPC-1(75%)	R-AD	CPA-1	EVCH(85%)R-ADEPC-1(75%)	
EAO-1(25%)			M (15%)	EAO-1(25%)
25.81%	16.13%	6.45%	9.68%	16.13%25.81%

Ex. 16				
EPC-1(75%)	R-AD	CPA-1	EVCH(85%)R-ADEPC-1(75%)	
EAO-1(25%)			M (15%)	EAO-1(25%)
24.24%	15.15%	12.90%	9.09%	15.15%24.24%

Ex. 17				
EPC-1(75%)	R-AD	CPA-3	EVCH(85%)R-ADEPC-1(75%)	
EAO-1(25%)			M (15%)	EAO-1(25%)
25.00%	15.63%	9.37%	9.37%	15.63%25.00%

Ex. 18				
EPC-1(75%)	R-AD	CPA-1(70%)	EVCH(85%)R-ADEPC-1(75%)	
EAO-1(25%)		CPA-4(30%)	M (15%)	EAO-1(25%)
24.24%	15.15%	12.90%	9.09%	15.15%24.24%

Ex. 19				
EPC-1(75%)	R-AD	CPA-4(50%)	EVCH(85%)R-ADEPC-1(75%)	
EAO-1(25%)		CPA-1(50%)	M (15%)	EAO-1(25%)
25.00%	15.63%	9.37%	9.37%	15.63%25.00%

Ex. 20				
EPC-1(75%)	R-AD	CPA-4(70%)	EVCH(85%)R-ADEPC-1(75%)	
EAO-1(25%)		CPA-1(30%)	M (15%)	EAO-1(25%)
25.00%	15.63%	9.37%	9.37%	15.63%25.00%

Ex. 21				
EPC-1(75%)	R-AD	PA-2(60%)	EVCH(85%)R-ADEPC-1(75%)	
EAO-1(25%)		CPA-4(40%)	M (15%)	EAO-1(25%)
25.81%	16.13%	6.45%	9.68%	16.13%25.81%

Ex. 22				
EPC-1(75%)	R-AD	PA-2(60%)	EVOH(85%)R-ADEPC-1(75%)	
EAO-1(25%)		CPA-4(40%)	M (15%)	EAO-1(25%)
24.24%	15.15%	12.90%	9.09%	15.15%24.24%
Ex. 23				
EPC-1(75%)	R-AD	CPA-3(90%)	EVOH(85%)R-ADEPC-1(75%)	
EAO-1(25%)		APA(10%)	M (15%)	EAO-1(25%)
24.24%	15.15%	12.90%	9.09%	15.15%24.24%
Ex. 24				
EPC-1(75%)	R-AD	CPA-2(90%)	EVOH(85%)R-ADEPC-1(75%)	
EAO-1(25%)		APA(10%)	M (15%)	EAO-1(25%)
25.81%	16.13%	6.45%	9.68%	16.13%25.81%
Ex. 25				
EPC-1(75%)	R-AD	CPA-2(90%)	EVOH(85%)R-ADEPC-1(75%)	
EAO-1(25%)		APA(10%)	M (15%)	EAO-1(25%)
24.24%	15.15%	12.90%	9.09%	15.15%24.24%
Ex. 26				
EPB(75%)	R-AD	CPA-3(40%)	EVOH(85%)R-AD EPB(75%)	
EAO-1(25%)		CPA-1(60%)	M (15%)	EAO-1(25%)
24.24%	15.15%	12.90%	9.09%	15.15%24.24%
Ex. 27				
EPC-2(75%)	R-AD	CPA-3(40%)	EVOH(85%)R-ADEPC-2(75%)	
EAO-1(25%)		CPA-1(60%)	M (15%)	EAO-1(25%)
24.24%	15.15%	12.90%	9.09%	15.15%24.24%
Ex. 28				
EPC-2(75%)	R-AD	CPA-4	EVOH R-ADEPC-2(75%)	
EAO-1(25%)			EAO-1(25%)	
23.33%	16.66%	13.33%	6.66%	16.66%23.33%
Ex. 29				
EPC-2(75%)	R-AD	CPA-4(70%)	EVOH R-ADEPC-2(75%)	
EAO-1(25%)		CPA-1(30%)	M (15%)	EAO-1(25%)
23.33%	16.66%	13.33%	6.66%	16.66%23.33%
Ex. 30				
EPB(75%)	R-AD	CPA-4(70%)	EVOH R-AD EPB(75%)	
EAO-1(25%)		CPA-1(30%)	M (15%)	EAO-1(25%)
22.67%	17.00%	12.99%	6.17%	16.37%22.67%
Ex. 31				
EPB(75%)	R-AD	CPA-4	EVOH R-AD EPB(75%)	
EAO-1(25%)			EAO-1(25%)	
23.12%	16.87%	12.50%	6.25%	16.25%23.12%
Ex. 32				
EPB(75%)	R-AD	CPA-4	EVOH R-AD EPB(75%)	
EAO-1(25%)			EAO-1(25%)	
27.78%	13.89%	11.11%	5.55%	13.89%27.78%



Ex. 33				
EPC-2(60%)	R-AD	CPA-1(50%)	EVCH	R-ADEPC-2(60%)
EAO-1(40%)		CPA-4(50%)		EAC-1(40%)
26.44%	14.61%	7.65%	6.75%	15.21% 29.23%
Ex. 34				
EPC-2(60%)	R-AD	PA-1(90%)	EVCH	R-ADEPC-2(60%)
EAO-1(40%)		CPA-1(10%)		EAC-1(40%)
26.44%	14.61%	7.65%	6.75%	15.21% 29.23%

In the foregoing Examples the following materials were employed:

EPC-1 Fina 8473 sold by Fina, a propylene/ethylene copolymer with 3.1% ethylene.

EPC-2 Eltex P KS 409 sold by Solvay, a propylene ethylene copolymer with 3.2% ethylene.

EPB W531D sold by Sumitomo, an ethylene propylene butene copolymer.

R-AD an ethylene alpha-olefin copolymer, Tymor(TM) 1203 sold by Morton Intl., rubber and maleic anhydride grafted.

P-AD Bynel E302 sold by DuPont, an anhydride grafted polypropylene.

PA or PA-1 8309 FN sold by Allied, Nylon 6.

PA-2 Vestamid L1940 Natural sold by Huls America, Inc. contains Irganox 1098, an antioxidant, and calcium stearate.

APA Sellar PA 3426 sold by DuPont, an amorphous nylon.

CPA-1 XE 3303 sold by Emser, a Nylon 6 6/6 10.

CPA-2 CF62BSE sold by Emser, a Nylon 6/6 9.

CPA-3 Ultramid C35 sold by BASF, a Nylon 666.

CPA-4 Grilon CF6S sold by Emser, a Nylon 6/12.

EVOH	Ethylene vinyl alcohol copolymer, Eval LC-E105A sold by Eval of America.
EVOH-2	ECG-156B sold by Eval of America, an ethylene vinyl alco- hol copolymer.
M	an ethylene - acrylic ester - maleic anhydride copolymer modi- fier.
EAO-1	Tafmer A-4085 sold by Mitsui, a homogeneous ethylene butene copolymer, density = 0.885 g/cc.

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EAO-1	Lower density ethylene alpha-olefin copolymer ATTANE(TM) 4203, sold by Dow Chemical, ethylene polymerized with 1- octene having a density of 0.905 g/cc
EAO-2	ethylene 1-octene copolymer, Dowlex(TM) 2045 sold by Dow Chemical
EBA	EA 705-009, sold by Quantum Chemical Company, ethylene butyl acrylate copolymer having a butyl acrylate content of 5% by weight.
COLOR-1	white color concentrate, EPE 10214-C sold by Teknor Color, a 50% TiO <sub>2</sub> /50% LDPE blend.

The foregoing description of preferred embodiments of the invention have been presented for purposes of illustration and description. It is not intended to be exhaustive or to limit the invention to the precise form disclosed, and modifications and variations are possible in light of the above teachings or may be acquired from practice of the invention. The embodiments were chosen and described in order to explain the principles of the invention and its practical application to enable one skilled in the art to utilize the invention in various embodiments and with various modifications as are suited to the particular use contemplated. For example, although backseamed casings are discussed herein, thermoplastic films employing the present seal layer and other structural layers are also within the scope of the present invention. It is intended that the scope of the invention be defined by the claims appended hereto, and their equivalents.



THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A heat-shrinkable multilayer film comprising a heat sealing layer comprising a blend of from about 50% to about 95% by weight of a propylene-based copolymer and from about 5% to about 50% by weight of a homogeneous ethylene alpha-olefin copolymer having a density below about 0.90 g/cc.
2. A heat-shrinkable multilayer film as set forth in claim 1 wherein said heat sealing layer comprises a blend of from about 70% to about 85% by weight of said propylene-based copolymer and from about 15% to about 30% by weight of said homogeneous ethylene alpha-olefin copolymer.
3. A heat-shrinkable multilayer film as set forth in claim 1 wherein said propylene-based copolymer is a copolymer of propylene and one or more other alpha-olefins.
4. A heat-shrinkable multilayer film as set forth in claim 3 wherein said propylene-based copolymer is a copolymer of propylene and ethylene.
5. A heat-shrinkable multilayer film as set forth in claim 4 wherein said propylene-based copolymer is a copolymer of propylene, ethylene and butene.
6. A heat-shrinkable multilayer film as set forth in claim 4 wherein said propylene-based copolymer comprise from about 94% to about 99% by weight of propylene and from about 1% to about 6% by weight of ethylene.
7. A heat-shrinkable multilayer film as set forth in claim 5 wherein said propylene-based copolymer comprises from about 90% to about 99% by weight of propylene, from about 1% to about 6% by weight of ethylene and from about 1% to about 10% by weight of butene.





8. A heat-shrinkable multilayer film as set forth in claim 1 further including an outer layer heat sealable to said heat sealing layer.

9. A heat-shrinkable multilayer film as set forth in claim 1 further including at least one internal layer containing a polyamide, a copolyamide or blends thereof.

10. A heat-shrinkable multilayer film as set forth in claim 9 wherein said polyamide is amorphous.

11. A heat-shrinkable multilayer film as set forth in claim 1 further including a barrier layer.

12. A heat-shrinkable multilayer film as set forth in claim 1 further including one or more adhesive tie layers.

13. A heat-shrinkable film as set forth in claim 1 wherein said film is corona treated.

14. A heat-shrinkable multilayer film comprising an inner heat sealing layer comprising a blend containing from about 5% to about 50% by weight of a homogeneous ethylene alpha-olefin copolymer having a density of below about 0.90 g/cc and an outer heat sealing layer comprising a blend containing from about 5% to about 50% by weight of a homogeneous ethylene alpha-olefin copolymer having a density of below about 0.90 g.cc.

15. A heat-shrinkable multilayer film as set forth in claim 14 further including at least one internal layer containing a polymeric material selected from the group consisting of polyamides, copolyamides, amorphous polyamides and blends thereof.

16 A multi-layer film substantially as hereinbefore described with reference to the Examples

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DATED this SIXTH day of AUGUST 1996

10 W. R. Grace & Co. - Conn.

by DAVIES COLLISION CAVE  
Patent Attorneys for the applicant(s)



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ABSTRACT OF THE DISCLOSURE

A multilayer heat-shrinkable film is provided which includes at least one heat sealing layer which is a blend of a propylene copolymer and a homogeneous ethylene alpha-olefin copolymer having a density below about 0.90 g/cc. Preferably, the outermost layer is compatible with the heat sealing layer for ready formation of a lap seal for end use applications such as backseamed casings. An internal polyamide-containing layer provides excellent elastic recovery.



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